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Materials Investigation and Tests for the Development of Space Compatible Electrical Connectors

Final Report
Phase 2, Task IV,
Elastomeric Seal
Materials

February 1 through
October 29, 1971

MSFC Contract
NAS8-26054

Prepared for:
National Aeronautics and
Space Administration
George C. Marshall
Space Flight Center
Huntsville, Alabama

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Report

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Phase 2 Task IV
Elastomeric Seal Materials
(February 1, 1971 through October 29, 1971)

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Final Report - Phase 2

Task IV

Elastomeric Seal Materials

1.0 Introduction

1.1 The purpose of this investigation was the selection of an elastomeric material suitable for use in electrical connector sealing members for space environments. Such members include wire sealing grommets as well as main-joint and interfacial seals and would be expected to function in the same manner as conventional materials. In addition, these parts must also be capable of withstanding temperatures from -200°C to +200°C, must be able to endure space vacuums with negligible outgassing, and must be non-flammable in pure oxygen atmospheres.

1.2 The initial phase of this investigation consisted of a literature survey which revealed that the best selection would appear to be from compounds based on copolymers of highly fluorinated olefins. The basic materials are produced by duPont as Viton and by the 3M Company as Fluorel.

1.3 During the initial phase five of these compounds were selected for molding evaluation:

a. duPont Viton VS-2001
b. Mosites Fluorel 1087-JJ
c. Raybestos Manhattan L-2231 (L-3217)
d. Raybestos Manhattan L-3583-2
e. Raybestos Manhattan L-4034-25

1.4 Also selected at the same time for molding evaluation was a flame retardant silicone described by Arthur D. Little, Inc. in their final report under Contract No. NAS 9-9510, dated July, 1970.

1.5 Phase 2 of our investigation was concerned primarily with a molding study of these materials. Electrical connector grommets come in a variety of sizes and arrangements and provide a number of molding problems. Most of these are related to the holes which seal around the individual wires.

1.6 The typical grommet hole includes a number of sealing webs which allow use of various diameter wire insulation in the same hole. These webs are created by undercuts in the mold pins which form the hole, and their withdrawal from the part stretches the rubber to the point where any weakness at the knit line can result in a split web.

1.7 The first quarterly report of Phase 2 covered the initial molding of these materials in (a) a compression set button mold, (b) a mold with straight-through holes (ref. fig. 1) and, (c) a mold with single webs on each pin (ref. fig. 2).

1.8 The second quarterly report covered the more difficult molding of grommets with two (ref. fig. 3) and three (ref. fig. 4 & 5) webs in each hole in the 16, 20,and 24 hole sizes. Also covered was the maintenance aging of the various sizes of holes by repeated insertions and removal of contacts.

1.9 The work done during the first two quarters indicates that duPont VS2001 and Raybestos Manhatton L-4034-25 can be molded into grommets having size 20 or larger holes conforming to our preferred web designs. The other three fluorinated olefin type materials were eliminated from consideration because of their comparatively poor moldability.

1.10 This final report covers the third quarter studies consisting of molding of the duPont VS-2001 fluoro-elastomer and of the flame resistant Arthur D. Little company's silicone in the size 22 and 24 holes with three webs in each hole. Both single cavity and four cavity molds were used. Also covered in this report are dielectric strength, arc resistance, Bashore rebound, and maintenance aging tests on the various materials that have been successfully molded.

2.0 Report

2.1 A four cavity JTRE type production mold with beryllium copper core pins was used for our initial work in this quarter. This was an 18-66 arrangement containing 66 size 22D holes in each cavity. The pins were removed, chrome plated, and replaced. All molding during this quarter was again done on 50 ton Hull transfer presses with variable injection speed control. Results obtained with this mold using duPont VS2001 are listed in Table I. Some distortion and blistering was obtained with this material even though the material was freshly catalyzed and had been freshened in a tight rubber mill shortly before molding. Since these results tend to verify the results obtained during the first two quarters, further molding of the VS-2001 was temporarily stopped.

2.2 At this time the molding effort was switched to an evaluation of the moldability of flame-resistant silicones as developed by Arthur D. Little, Incorporated. The specific polymer used was General Electric's SE-517 reinforced gum. The formulation used was the one recommended by Arthur D. Little, Inc. as giving the best overall balance of physical properties.

2.3 The flame retardant agent, decabromodiphenyl (DBDP) is made by Michigan Chemical Corporation but requires recrystallization by Arthur D. Little Incorporated before it can be successfully used. One sample was obtained from Arthur D. Little (lot #644-145-B) who made it up for us in their laboratory. Commercial quantities are not available.

2.4 In order to conserve the sample of DBDP, our initial molding trials were made on the silicone rubber composition with the DBDP omitted as follows:

G.E. SE517 (predried 4 hours at 400°F)	100 parts
Cabosil HS-5	8 parts
Cadox TS-50 (2,4 - dichlorobenzoyl peroxide)	1.2 parts

2.5 This material was molded in the four-cavity 18-66 arrangement with results as listed in Table II. Moldability was very good, encouraging us to proceed with material having the DBDP flame retardant added.

2.6 A new batch was, therefore, prepared in accordance with the following formula. Note that the ingredients must be added in the order listed.

G.E. SE517 (predried 4 hours at 400°F)	100 parts
DBDP	75 parts
Cabosil HS-5	8 parts
Cadox TS-50	1.2 parts

2.7 Although the same 18-66 arrangement mold was used, much more mold sticking was encountered after the DBDP was added than before. The use of various lubricants in an attempt to overcome this condition was unsuccessful. The results obtained are tabulated in Table III.

2.8 Working on the premise that the chrome plated beryllium copper pins might be causing the mold sticking condition, the single cavity 18-2 mold used in the second quarter with the duPont VS-2001 material was used to evaluate the flame resistant silicone formula given in para. 2.6. This mold contains 82 size 24 pins made of music wire. DuPont DFL lubricant was used for this molding. Table IV lists the molding conditions and results. The results indicate that this material can be molded into grommets with holes as small as size 24 in our standard, preferred web design.

2.9 Because of the encouraging results on the 18-2 mold with the flame resistant silicone it was decided to try this mold a second time with duPont VS2001, using various lubricants. Results, as indicated in Table V, are somewhat inconclusive. Frekote 33H, however, does appear to give a better release condition than the Contour 318 used previously with this material.

2.10 Another attempt was now made to mold VS2001 in the 18-66 arrangement but using Frekote 33H in place of the 318 Contour release previously used. The first load exhibited several cracked webs but no blisters. A second separately catalyzed batch of VS2001 immediately produced blistered, spongy parts with no cracked webs. Of the molding variables evaluated, only a change to a longer cure corrected the blistered condition. This longer cure time caused cracked webs when molding earlier batches. The results are listed in Table VI and indicate that the duPont VS2001 material can possibly be molded into grommets having size 22D or larger holes conforming to our standard JTRE web design.

2.11 These results and those obtained earlier indicate that there is a variation in duPont VS2001 from one catalyzed batch to the next. It also appears that aging and remilling of a particular catalyzed lot improves the moldability and increases the cure rate of that lot.

2.12 The good parts obtained during this quarter were post cured according to the following schedules:

Material	Original Durometer "A"	Final Cure Time(hrs.)	Final Cure Temp(°F)	Post Cured Durometer "A"
*SE517+DBDP	50-52	4	400	56-57
**VS-2001	51-55	48	450	55-58

*Preliminary step cure - 1 hr. at 300°F, 1 hr. at 350°F
**Preliminary step cure - 1 hr. at 250°F, 1 hr. at 300°F, 1 hr. at 350°F, 1 hr. at 400°F

2.13 The typical connector grommet must be capable of undergoing considerable stretching during assembly and must also seal around wires after assembly. These conditions require a lively, flexible elastomer As a means of comparing the different materials, Bashore Rebound tests were run on post cured samples using a Precision Scientific Company Bashore Resiliometer. Following are the average values for each material.

duPont VS-2001	-	7.0
R.M. L-4034-25	-	7.5
G.E. SE-517 + DBDP	-	43.0

2.14 The Bashore readings indicate that the flame-resistant silicone is more than adequate while the fluoro-elastomers are extremely "dead" stocks by comparison and only performance testing of actual connectors would prove their suitability where this property is important.

2.15 Samples of the post cured parts were subjected to the maintenance aging test. This test consists of inserting and removing the proper size contact ten times in each hole to be tested. Any cracking or splitting of the web is considered a failure. Table VII is a summary of the results obtained from the maintenance aging tests conducted on parts molded in this quarter.

2.16 The maintenance aging results obtained with VS2001 are somewhat improved compared to the results obtained during the second quarter testing. This may be due to close control of molding conditions and to more experience in molding this type of material. The results obtained should be adequate although they are decidedly poorer than the standard silicone materials now used.

2.17 The maintenance aging results obtained with the silicone DBDP combination indicate that this material is definitely superior to the fluoro-elastomers in resistance to repeated insertion and removal of contacts.

2.18 Electrical Properties: Selection of the materials for this evaluation was based on characteristics other than electrical properties. However, they still must be adequate for electrical connector requirements. Sample test specimens 1/8 inch thick by four inches diameter were therefore molded from VS2001, L-4034-25, and the flame retarded silicone rubber and tested for dielectric strength and arc resistance. Dielectric strength was tested under oil in accordance with MIL-M-14F. One sample of each material was tested with no special preconditioning by the short-time method. A second sample was tested, after immersion for 48 hours in water at 50°C, using the step by step method. Arc resistance was tested in accordance with MIL-C-5015D. Results are in Table VII and indicate all to be adequate electrically with the silicone material best in both categories.

3.0 Conclusions

3.1 DuPont VS2001 and Raybestos Manhattan L-4034-25 are superior to the other fluoro-elastomers tested from the standpoint of moldability and are essentially equal in this respect. With proper molds and close control of molding conditions, they appear capable of providing satisfactory parts in most standard density arrangements using size 20 contacts or larger. The higher density arrangements and those with smaller pins are more difficult to mold but in some cases molding may be possible.

3.2 Mold pins must be stainless steel or chromium plated. Beryllium copper pins do not provide adequate mold release for the fluoro-elastomers. Even so, stripper plates are necessary to remove parts from the mold if the part is a high density arrangement containing size 22 or smaller pins or if the part is greater than 0.340 inch thick.

3.3 The degree of uniformity of these materials from batch to batch is not known. The duPont VS2001 material varied in moldability from one catalyzed batch to the next. This caused difficulties in establishing a reproducible molding cycle and would have to be controlled before this material could be used in production. We have no assurance that R/ML-4034-25 is better for only one lot of material was used in our investigation.

3.4 The limited number of performance oriented tests conducted indicate these materials are adequate, though not exceptional, electrically and will withstand a limited amount of maintenance aging. Flammability in oxygen atmospheres has previously been established as satisfactory by NASA and the limited amount of outgassing information available indicated this to be acceptable. However, a considerable amount of additional testing would be necessary to indicate performance in a completed connector, especially because of the "dead" or non-elastic nature typical of those materials.

3.5 The flame retardant silicone materials developed by Arthur D. Little Incorporated appear promising. Moldability is superior to the fluoro-elastomers as are the electrical properties and resilience. The large amount of flame retarder added to impart flame resistance does take its toll in heat resistance and in mechanical properties. Again performance testing of actual connectors is absolutely necessary before its use could be justified.

3.6 Two other deterrents exist with this material. The flame retarder, decabromodiphenyl in the recrystallized condition required is not yet commercially available. Moreover, while it increases the flame resistance in oxygen atmosphere considerably over previous silicone rubber, the results are still somewhat short of NASA optimum requirements. This would necessitate some relaxation of the flame resistance requirements but it seems evident that a completely wired connector assembly should provide adequate resistance to combustion and flame propagation.

3.7 This investigation has as its main achievements the uncovering of three materials having improved flame resistance in oxygen-rich atmospheres and the development of technological capability whereby they can be molded into complex electrical connector grommets. The ability of these materials to meet all the performance requirements which electrical connectors must exhibit can only be proven by actual tests on the complete connectors themselves. The advances made thus far would certainly seem to warrant completion of such a program.

Table I

Fluoro-elastomer Molding Evaluation:
Triple Web Holes (18-66) - 4 Cavities
duPont VS-2001

Mold Temperature (°F)		Transfer Pressure (psi)	Fill Time (sec)	Cure Time (min)	Results
Original	Final				
270-295	300	800	9	2 3/4	Stuck on top web pins - 2 pieces torn
250-290	295-300	800	9-10	2 1/2	On top - came off without tearing - some distortion - small blisters
260-290	300-305	800	10-11	2 1/2	Slight improvement - 2 pieces on top, 2 on bottom - came off without tearing
280-290	305-310	800	18-19	2 1/2	All on top pins - cracked webs on 1 piece - some distortion - no blisters
280-295	310	800	18-19	2 1/4	All on top - less distortion small blisters around outside
290-305	310-320	800	18-19	2 1/4	3 on top, 1 on bottom - 1 piece blistered -others OK
300-310	320-325	800	18-19	2	All on top - 3 pieces partially split - no blisters
310-320	320-330	800	17-18	1 3/4	All on top - cracked webs - many small blisters
285-295	300	800	12-13	2 1/2	1 on top, cracked webs - 3 on bottom, cracked on bottom
290-295	300-310	800	28-30	2 1/2	All on bottom pins - 2 partially torn

Note: Contour 318 Mold Release used in all cases

Table II

Silicone SE517 Molding Evaluation:
Triple Web Holes (18-66) - 4 Cavities

Mold Temperature (°F)		Transfer Pressure (psi)	Fill Time (sec)	Cure Time (min)	Results
Original	Final				
225-245	260-265	300	6-7	10	All in cavity plate - ideal condition - brown gas marks on 1 piece - depression at gates (excess pressure)
225-240	260-265	150	6-7	10	3 in cavity plate - 1 on bottom pins peeled off OK - gas mark on side of 1 piece
225-245	260-265	100	6-7	10	Similar to above
230-235	250	100		8	Non-fill
225-235	250-255	180	25-30	8	All in cavity plate - OK
225-240	260	200	17-18	10	All in cavity plate - slight depression at gates - otherwise OK

Note: duPont DFL #3 Water Soluble Mold Release used in all cases

Table III

Flame Resistant Silicone Molding Evaluation:
Triple Web Holes (18-66) - 4 Cavities

Mold Temperature (°F)		Transfer Pressure (psi)	Fill Time (sec)	Cure Time (min)	Results
Original	Final				
235-240	260-265	200	25-27	10	75% fill - 3 pieces in cavities - 1 on bottom pins
230-245	260-265	250	18-20	10	All on bottom pins - peeled off with difficulty - small tears on 3 - some shredding in holes on all 4
225-245	260-265	250	17-18	10	Similar to previous shot (#2)
*230-240	255-260	250	15	10	Slight improvement on #3 above - 7 cracked webs near gates of 3 pieces - all pieces did not quite fill on bottom opposite gates
*225-250	260	250	17	10	Improvement on #4 above - 3 cracked webs along knit lines near gates (1 on each piece) - only 1 torn during peeling from pins - all 4 did not quite fill on bottom opposite gates.
**225-240	255-260	250	12-13	10	Similar to #4 and 5 above except 1 piece only had trace of porosity on bottom opposite gate - 4 cracked webs on 1 piece only

Note: duPont DFL#3 Release except where noted, otherwise
*DFL#3 on top pins, Contour 318 on bottom pins.
**DFL#3 on top pins, Frekote 33H on bottom pins.

Table IV

Flame Resistant Silicone Molding Evaluation:
Triple Web Holes (18-2) - Single Cavity

Mold Temperature (°F)		Transfer Pressure (psi)	Fill Time (sec)	Cure Time (min)	Results
Original	Final				
225-250	265	250	7-8	10	Stuck on bottom pins - outside cracked during peeling from pins - otherwise OK
240-250	265	200	6-8	10	Short fill - on bottom - peeled off OK
230-245	265-270	230	6-8	10	Partially lifted off bottom pins - difficult to peel but did not tear - small porous spot on bottom opposite gate - otherwise OK
235-240	260-265	240	12-15	10	Similar to previous piece - no porosity - OK
225-240	260-265	240	15-16	10	Similar to previous 2 pieces - no porosity OK

Note: duPont DFL#3 Mold Release used in all cases.

Table V

Fluoro-elastomer Molding Evaluation:
Triple Web Holes (18-2) - Single Cavity
duPont VS-2001

Mold Temperature (°F)		Transfer Pressure (psi)	Fill Time (sec)	Cure Time (min)	Results
Original	Final				
270-290	305-310	700	5-7	2 1/2	Partially lifted off bottom pins - tore during removal - no blisters or porosity
*280-290	300-305	650	8-9	2 1/2	Lifted partially - 2/3 tore off and stuck to pins
**280-295	305-310	600	10-11	2 1/2	On bottom pins - peels off slowly by hand - several cracks on bottom and in holes may be due to lube - 7 webs cracked
275-290	300-305	550	13-15	2 1/2	No lube on bottom - piece on bottom - top and outside diameter tore off - rest stuck to bottom pins

Note: duPont DFL#3 Release used except where noted
*DFL #3 on top pins, Contour 318 on bottom pins
**DFL #3 on top pins, Frekote 33H on bottom pins

Table VI

Fluoro-elastomer Molding Evaluation:
Triple Web Holes (18-66) - 4 Cavities
duPont VS-2001

Mold Temperature (°F)		Transfer Pressure (psi)	Fill Time (sec)	Cure Time (min)	Results
Original	Final				
285-305	305-310	750	8-9	2 1/2	Loose on bottom pins - 1 piece cracked on bottom - all with cracked webs - no blisters
280-310	300-305	700	9	2 1/2	Stayed on bottom pins - spongy - many blisters
285-305	305-310	700	9	2 1/2	Somewhat worse - 2 pieces tore off and stuck to bottom pins
275-295	300-305	700	8-9	2 1/2	Blistered - 3 cracked on bottom - 2 with bottom porosity
275-300	300-310	700	8	2 1/2	Slightly worse - more distortion caused by longer transfer cycle
275-310	305	700	12	2 1/2	Distorted - cracks on bottom -fewer blisters
275-305	310-315	700	11-12	5	Loose on bottom pins - can be lifted off much easier but care is required - 3 pieces cracked on bottom - no blisters or distortion
285-305	305-310	700	11-12	5	Lifts easily off bottom pins - small crack on hole edge - otherwise OK
285-305	310	700	10-11	5	Lifts easily off bottom pins - edge chip on 1 piece - otherwise OK

Notes: Frekote 33H mold release used in all cases.
First load made from material catalyzed 8/31/71
All other loads made from material catalyzed 9/13/71

Table VII

Maintenance Aging
Triple Webs

Material	Arrangement	Hole Size	Results
VS-2001	18-66	22D	*3 pieces - 1 of 9 holes failed - second insertion
Silicone +DBDP	18-2	24	3 pieces - 9 holes - no failures -some scuffing on surface and edge of web

*Failure at knit line opposite gate - last spot to fill in cavity

Table VIII

Electrical Tests

Dielectric Strength:	Viton VS-2001	Fluorel L-4034-25	Silicone + DBDP
Short-Time Method: Stepwise Method, D48/50:	280 vpm 244 vpm	264 vpm 268 vpm	400 + vpm 372 vpm
Arc Resistance, Seconds:	121 sec	127 sec	180 + sec

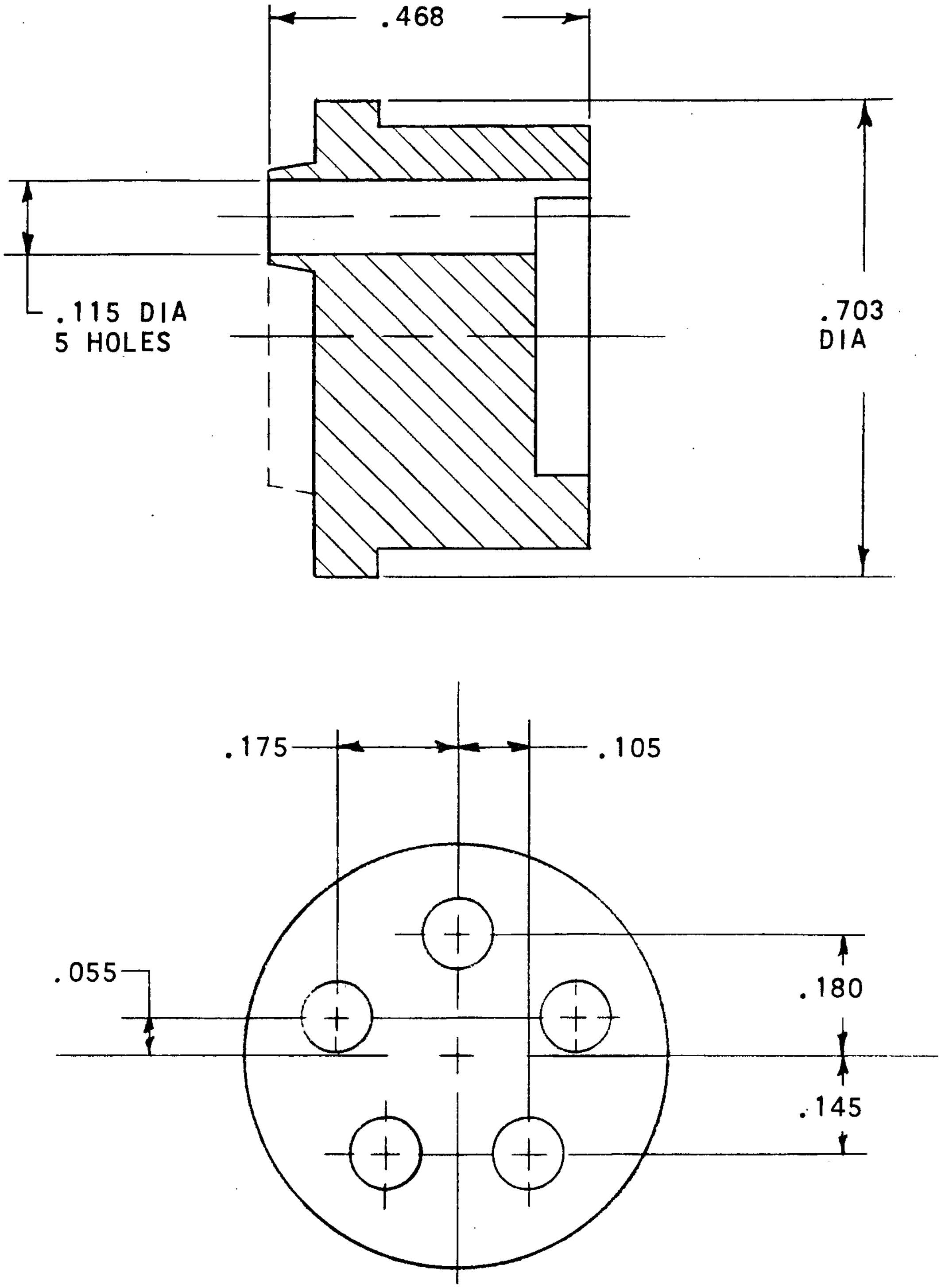


Figure 1.

Typical Dimensions of Samples Used to
Evaluate Molding of Straight-Through Holes.

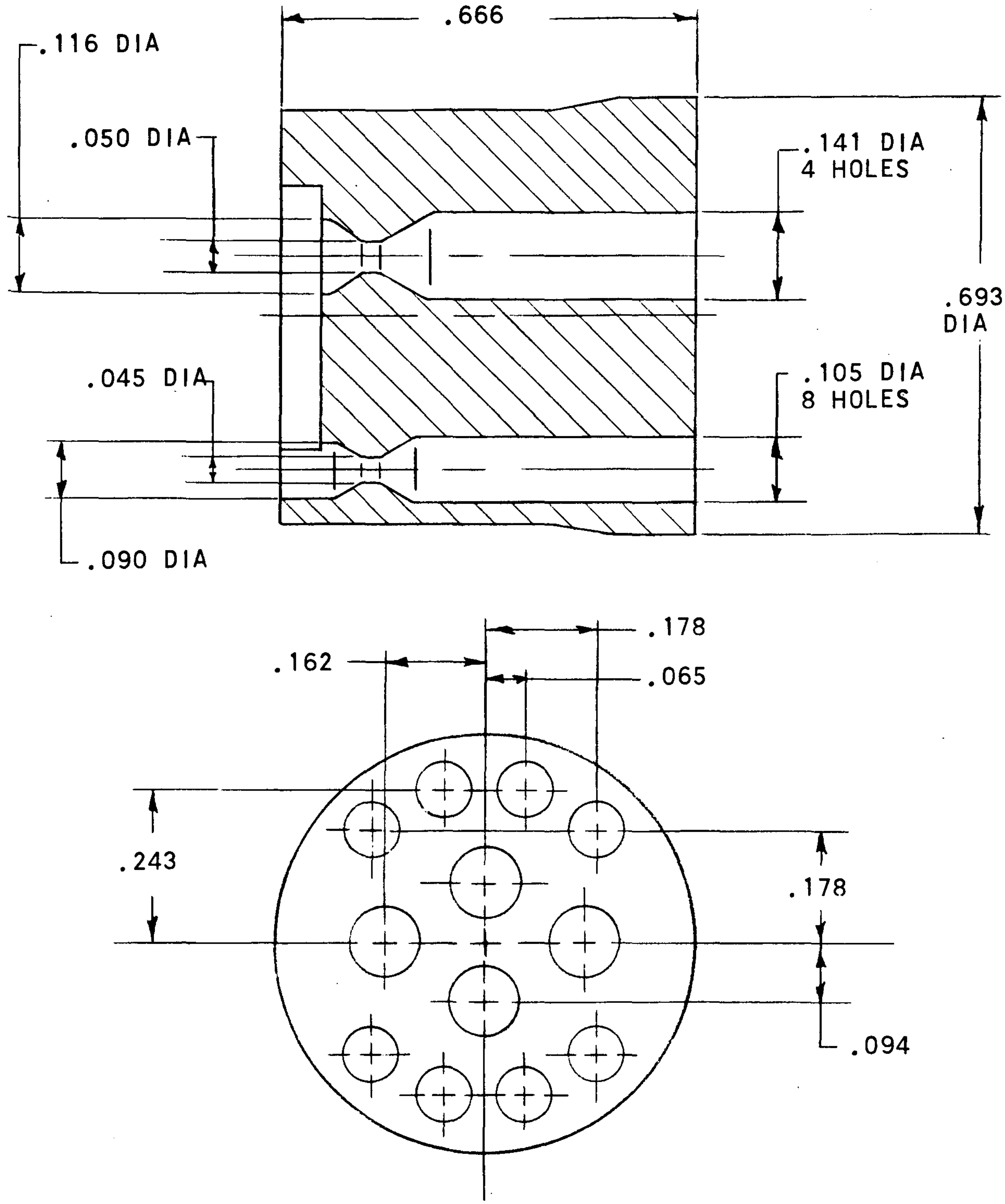


Figure 2.

Typical Dimensions of Samples Used to Evaluate Molding of Single Web Holes.

ARRANGEMENT	NUMBER OF HOLES	TYPICAL SPACING	A DIA	B DIA	C DIA
22-21	21	.188	.044	.155	.054
22-55	55	.130	.038	.105	.045

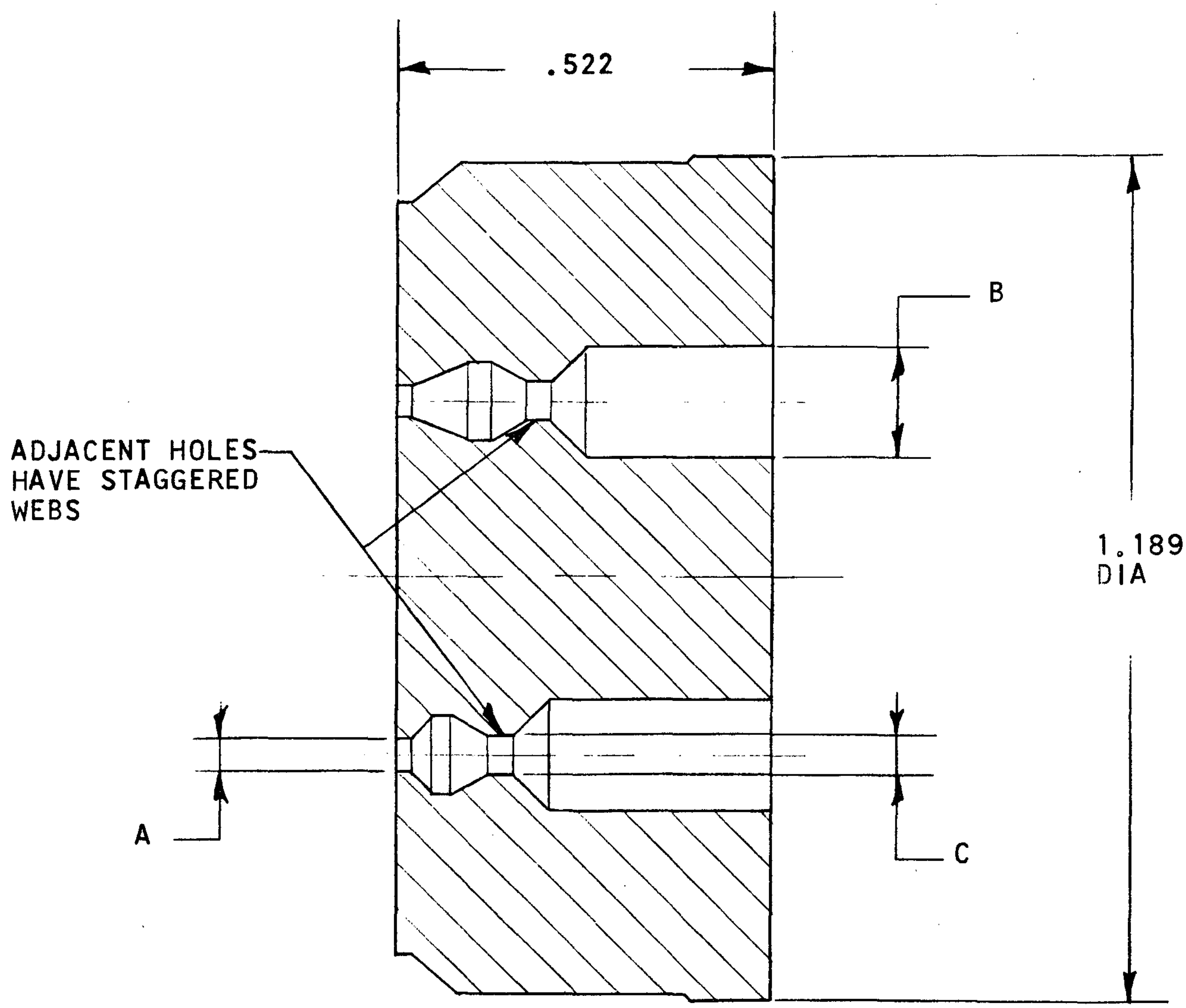


Figure 3.

Typical Dimensions of Samples Used to Evaluate Molding of Double Web Holes.

ARRANGEMENT	NUMBER OF HOLES	TYPICAL SPACING	A DIA	B DIA	C DIA
18-2	82	.082	.015	.060	.015
18-11	11	.191	.034	.124	.029
18-66	66	.090	.022	.068	.022

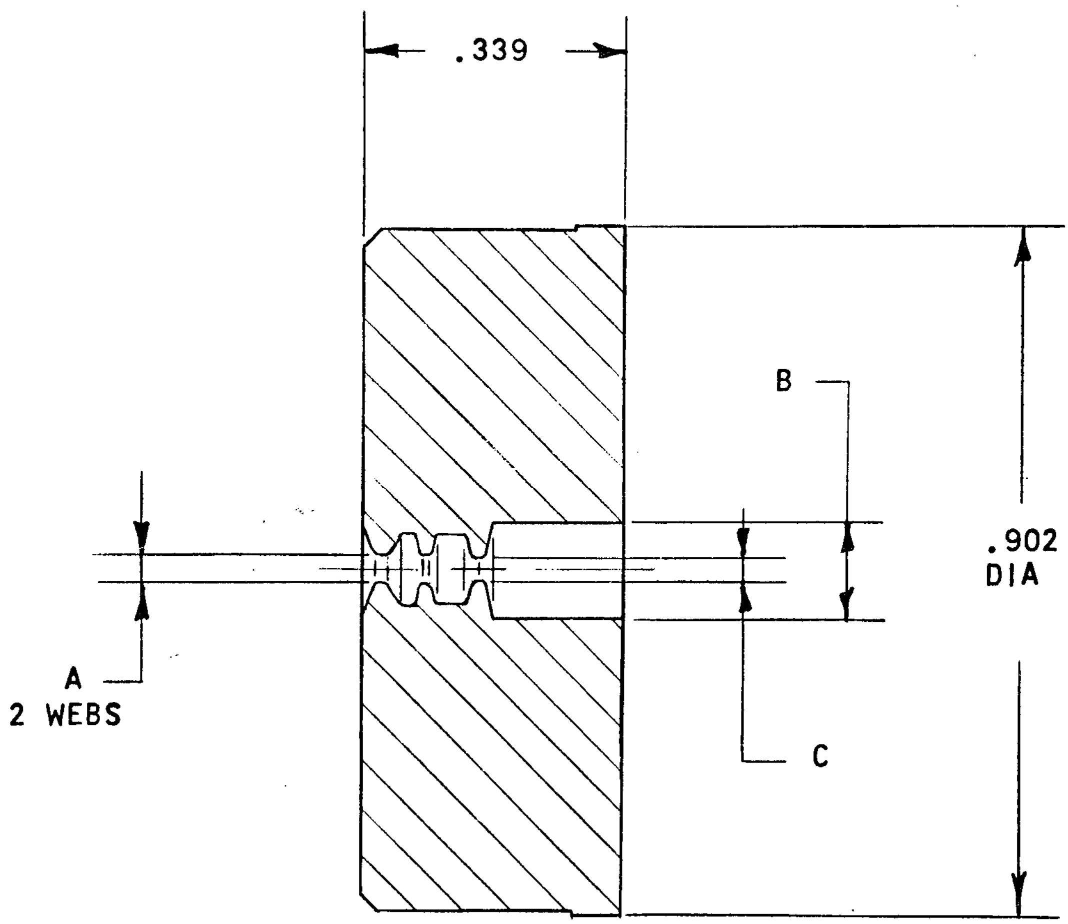


Figure 4.

Typical Dimensions of Samples Used to Evaluate Molding of Short Triple Web Holes

ARRANGEMENT	NUMBER OF HOLES	TYPICAL SPACING	A DIA	B DIA
22-21	21	.188	.051	.124
22-55	55	.130	.030	.103

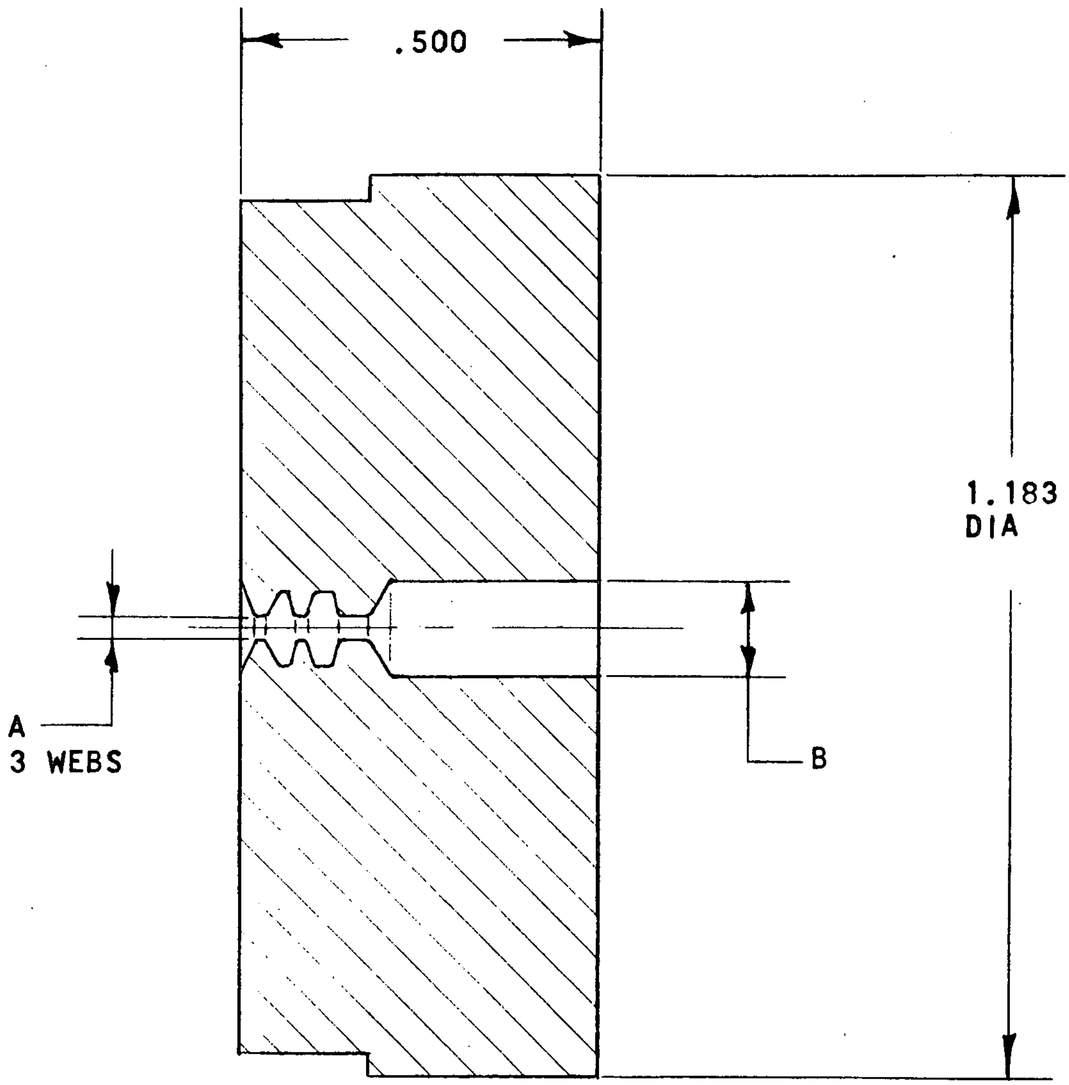
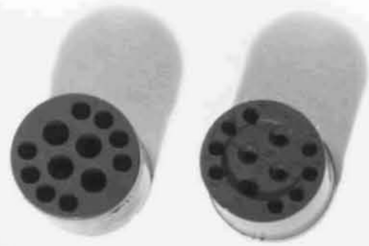
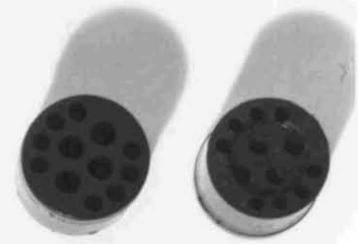


Figure 5.

Typical Dimensions of Samples Used to Evaluate Molding of Long Triple Web Holes

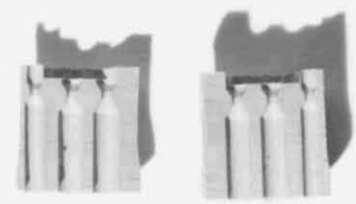


RAYBESTOS MANHATTAN
L-4034-25
Single Web Holes
Post Cured



DUPONT VS2001
Single Web Holes
Post Cured

DUPONT VS2001
Single Web Holes
As Molded



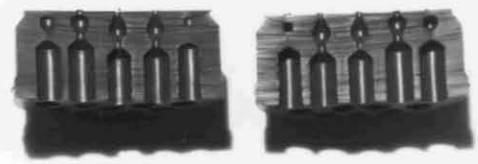
RAYBESTOS MANHATTAN
L-4034-25
Single Web Holes
As Molded

RAYBESTOS MANHATTAN
L-2231
Straight Through Holes
Post Cured

DUPONT VS2001
Straight Through Holes
As Molded

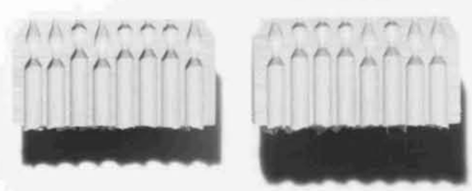
Figure 6.

L-4034-25 Post Cured
22-21
Size 16 Holes
Double Webs



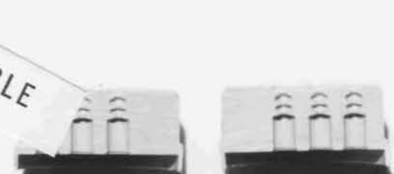
L-2231
22-21
Size 16 Holes
Double Webs

VS-2001 Post Cured
22-55
Size 20 Holes
Double Webs



L-4034-25
22-55
Size 20 Holes
Double Webs

VS-2001 Post Cured
18-11
Size 16 Holes
Size 20 Triple Webs



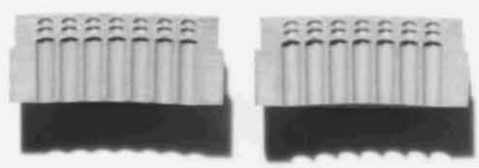
L-4034-25
18-11
Size 16 Holes
Size 20 Triple Webs

Figure 7.

VS-2001 Post Cured
22-21
Size 16 Holes
Triple Webs

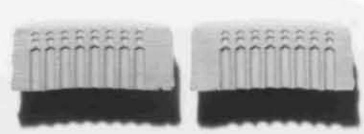
L-4034-25
22-21
Size 16 Holes
Triple Webs

22-55
Size 20 Holes
Triple Webs



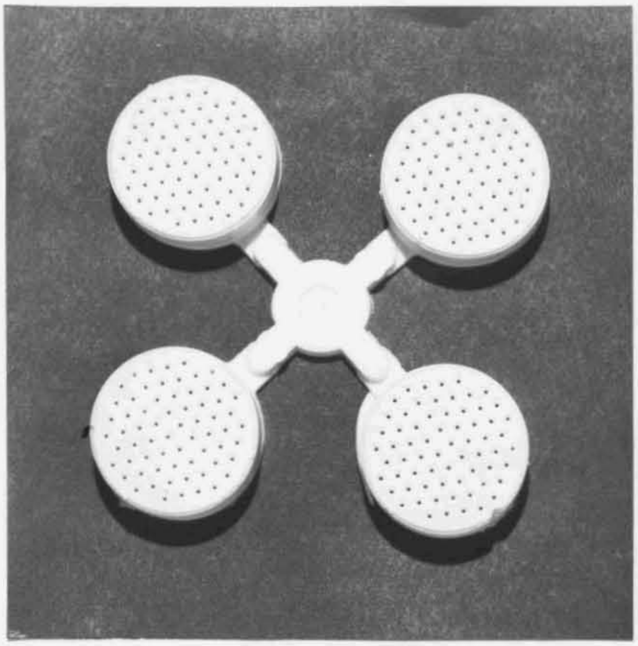
VS-2001
22-55
Size 20 Holes
Triple Webs

L-4034-25
18-2
Size 24 Holes
Triple Webs

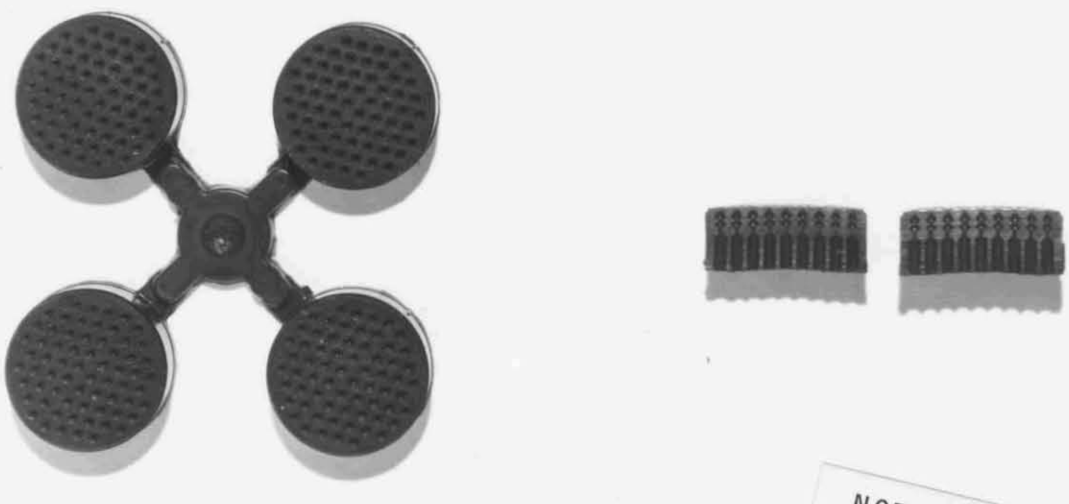


L-4034-25
18-2
Size 24 Holes
Triple Webs

Figure 8.



SE517 + DBDP
18-66
SIZE 22D HOLES
TRIPLE WEBS



VS-2001 POST CURED
18-66
SIZE 22D HOLES
TRIPLE WEBS

Figure 9.